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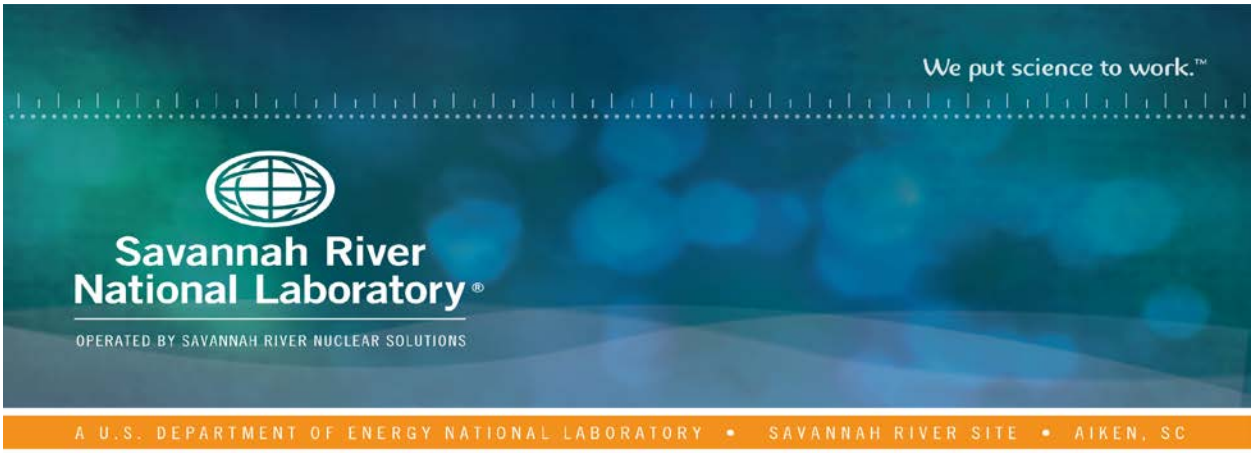

Saltstone Third Quarter Calendar Year 2019 (3QCY19) Toxicity Characteristic Leaching Procedure (TCLP) Results

K. A. Hill

February 2020

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EXECUTIVE SUMMARY

The aqueous waste from Tank 50 (salt solution) is sampled quarterly for transfers to the Saltstone Production Facility (SPF). Salt solution is treated at SPF and disposed of in the Saltstone Disposal Facility (SDF). Per request of customer, X-TAR-Z-00008, Revision 0[1], two SDF waste form (saltstone) samples were prepared in the Savannah River National Laboratory (SRNL) from the Tank 50 Waste Acceptance Criteria (WAC) sample and Z-Area premix material for the third quarter of calendar year 2019 (3QCY19).[2,3] One sample contained a Full Premix which included 10:45:45 (by weight) of cement, slag and fly ash.[1] The second sample contained 60:40 (by weight) of slag and fly ash only referred to as the "Cement-Free grout sample".[1] Results from this technical report support Task 2: 'Grout Leaching Analyses' of the Task Technical Request (TTR)[3] prepared by Savannah River Remediation (SRR). After a 28 day cure, a sample of each of the SDF waste forms was collected and shipped to a certified laboratory for analysis using the Toxicity Characteristic Leaching Procedure (TCLP).[4] The 3QCY19 saltstone (Full Premix) and the Cement-Free grout samples met the South Carolina (SC) Code of Regulations for Hazardous Waste Management Regulations (HWMR) 61-79.261.24 and 61-79-268.48 requirements for a non-hazardous waste form with respect to Resource Conservation and Recovery Act (RCRA) metals and Underlying Hazardous Constituents (UHCs), and also met the SPF WAC.[5-7]

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LIST OF ABBREVIATIONS

ARP/MCU	Actinide Removal Process / Modular Caustic Side Solvent Extraction Unit
D&S-FE	DWPF & Saltstone Facility Engineering
DSSHT	Decontaminated Salt Solution Hold Tank
EC&ACP	Environmental Compliance & Area Completion Projects
EM&ES	Environmental, Materials & Energy Sciences
EPA	Environmental Protection Agency
ETF	Effluent Treatment Facility
LOD	Limit of Detection
LOQ	Limit of Quantitation
MRL	Minimum Reporting Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NRC	Nuclear Regulatory Commission
RCRA	Resource Conservation and Recovery Act
RPD	Relative Percent Difference
RL	Reporting Limit
SDF	Saltstone Disposal Facility
SPF	Saltstone Production Facility
SRNL	Savannah River National Laboratory
SRNS	Savannah River Nuclear Solutions
SRR	Savannah River Remediation
SwRI®	Southwest Research Institute
TCLP	Toxicity Characteristic Leaching Procedure
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
UHC	Underlying Hazardous Constituents
WAC	Waste Acceptance Criteria

1.0 Introduction

The SPF receives waste from Tank 50 for treatment. The following dates were selected starting from the last quarterly sampling date to the current quarterly sampling date. Tank 50 accepted the following transfers from May 8, 2019 (when it was ~44% full) to August 6, 2019 (when it was ~57% full):[8] During this same time period there was a total of 7.5 kgal of Tank 50 material transferred out to Z Area.

- ~3.2 kgal from 211-H
- ~12.0 kgal from Effluent Treatment Facility (ETF)
- ~85.6 kgal from the Actinide Removal Process / Modular Caustic Side Solvent Extraction Unit (ARP/MCU) Decontaminated Salt Solution Hold Tank (DSSHT)
- ~21.3 kgal from 512-S
- ~8.2 kgal from LWHT and flush water

On August 6, 2019, a salt solution sample was taken from Tank 50[9] and later used to prepare two SDF waste form samples, referred to as a Full Premix saltstone sample and Cement-Free grout sample.[10] The Full Premix sample is the baseline, historical formulation for saltstone and is the sample used to determine that saltstone produced by the SPF is a non-hazardous waste form. The Cement-Free sample is included as a preliminary examination of the potential use of a Cement-Free formulation in future saltstone processing. The 3QCY19 Full Premix and Cement-Free saltstone samples were prepared on August 28, 2019.[11] The 3QCY19 salt solution had been stored since collection in a zero-headspace Teflon® bottle refrigerated at <10 ºC to preserve the mercury species present in the sample. Once the 3QCY19 saltstone samples cured for 28 days, they were crushed, sieved, packaged, and deemed "collected".[12] The samples were then shipped to Southwest Research Institute (SwRI®) to analyze for toxicity per the TCLP method.[2,4] The full premix saltstone sample determines whether the non-hazardous nature of the grout meets the requirements of the SC Code of Regulations 61-79.261.24[6] for RCRA metals and 61-79.268.48[5] for inorganic/organic UHCs (for informational purposes only[3]).

2.0 Experimental

Saltstone preparation was performed at SRNL. DWPF & Saltstone Facility Engineering (D&S-FE) provided SRNL with the saltstone grout recipe as well as the premix components.[3] Table 2-1 shows the premix components obtained to date for CY2019 samples with specific LOT numbers.[11] SRR directed SRNL personnel to use the same premix components for the 3QCY19 saltstone samples as were previously used for the 2QCY19 saltstone samples.[11]

Table 2-1. Premix Components for CY2019

Premix Component	Date SRNL Received	LOT #
Holcim Cement 3QCY19	6/27/2019	2019-IR-05-0487
Lehigh Slag 3QCY19	6/27/2019	2019-IR-05-1040
SE Fly Ash 3QCY19	6/27/2019	2019-IR-05-0714
Holcim Cement 2QCY19	6/27/2019	2019-IR-05-0487
Lehigh Slag 2QCY19	6/27/2019	2019-IR-05-1040
SE Fly Ash 2QCY19	6/27/2019	2019-IR-05-0714
Holcim Cement 1QCY19	3/04/2019	2019-IR-05-1666
Lehigh Slag 1QCY19	3/04/2019	2019-IR-05-0120
SE Fly Ash 1QCY19	3/04/2019	2019-IR-05-0195

The saltstone samples were prepared using the mixing method outlined in SRNL Environmental, Materials & Energy Sciences (EM&ES) work instructions and the 3QCY19 premix components in Table 2-1.[13] The samples cured in a Ziploc® sealed plastic bag for 28 days. After curing, the samples were crushed and sieved using the method outlined in EM&ES work instructions.[14] Material that passed through the 3/8-inch (0.375") sieve (9.252 mm) was subsequently screened through a No. 4 sieve (4.76 mm). The material retained on the No. 4 sieve was packaged in a primary container (250 mL High Density Polyethylene (HDPE) bottle) and shipped on the same day that it was prepared to SwRI® by Environmental Compliance & Area Completion Projects (EC&ACP).[3]

3.0 Results

Table 3-1 summarizes the analytical results provided by the vendor, SwRI.[15] The first eight rows show data for the RCRA metals and the next four rows show data for the UHC metals from the TCLP leachates. The last four rows show results from solids analyses of the saltstone for benzene, phenol, total and amenable cyanide. The entire vendor report is documented and included as a reference.[15] Some of the data values are flagged with qualifier letters (U, L, D, J, B) that are shown as footnotes to the table. Further explanation for these qualifiers can be found in the vendor report.[15] For comparison, the previous quarter and four quarter average results for the Full Premix sample are shown. The four-quarter average values contain a qualifier (*, +, ^) if past values have been reported as a non-detectable analyte ('U'). The Regulatory Toxicity[6] values and the WAC Limits are from Table 6 of the WAC[7] and reflect the requirements in the applicable version of the document. Note that the vendor used a "modified" Method 1311 where sample mass was restricted due to the elevated activity of the sample.[15] This methodology is consistent with the joint guidance from the Nuclear Regulatory Commission (NRC) and Environmental Protection Agency (EPA) for mixed radioactive and hazardous waste.[16] For 3QCY19 the TCLP extraction was performed on both the Full Premix and the Cement-Free samples using extraction fluid #2 which is an aqueous acetic

acid solution with pH= 2.88 ± 0.05 prepared by diluting 5.7 mL of glacial acetic acid into reagent water for a total volume of 1 Liter.[15]

Full-Premix Saltstone

Table 3-1 shows the reported detection limit for As has remained the same (<0.025 mg/L) relative to the previous quarter. The reported value for Se of <0.025 mg/L is below the LOD of 0.025 mg/L. Lead has remained as less than detectable at <0.0075 mg/L in comparison to the previous quarter. The analyzed value for Be of <0.005 mg/L is the same as the previous quarter. The reported value for Cr for this quarter was measured at <0.005 mg/L and is similar to the previous quarter. The reported TCLP value for Ba of 0.45 mg/L is comparable to the previous quarter.

The mercury TCLP value for the 3Q19 sample of 0.0016 mg/L is lower than the previous quarter measuring 0.0036 mg/L. Total mercury in the Tank 50 WAC samples were similar at 63.5 mg/L for 2Q19 and 67.6 mg/L for 3Q19.[17] Mercury speciation analyses for recent past 3Q17 through 3Q19 show that the total mercury levels in the Tank 50 supernate have ranged from a low of 61.7 mg/L for 4Q18 to a high of 81.4 mg/L for the 3Q17 sample as shown in Table 3-2.[18] The corresponding methyl Hg values expressed as mg Hg/L ranged from 18.8 mg/L to 36.6 mg/L. The methyl Hg species is the dominant Hg species in the Tank 50 supernate (relative to other Hg species measured like elemental Hg(0) or ionic Hg(I) and/or Hg(II)) with methyl Hg to total Hg ratios shown in Reference [19] that are in the range of 0.29 to 0.51.

Cement-Free Saltstone

All of the RCRA metals and UHCs shown in Table 3-1 for the Full Premix and Cement-Free samples are comparable, i.e., within 50% of the value. The amenable and total cyanide and phenol for the Cement-Free sample are higher than for the Full Premix sample. The cement-free values for total cyanide and phenol are also higher than the four-quarter average of the Full Premix.

Table 3-1. 3QCY19 Saltstone Sample TCLP and Solids Analysis Results

Analyte	Full Premix Result (mg/L)[15]	Cement-Free Result (mg/L)[15]	Regulatory Toxicity[6] (mg/L)	WAC Limit[7] (mg/L)	Results Full Premix	
					Previous Quarter[20] (mg/L)	Previous Four Quarter Average[20-23] (mg/L)
RCRA Metals						
Arsenic (As)	<0.025[U]	<0.025[U]	5.0	2.5	<0.025[U]	0.029[+]
Barium (Ba)	0.450[D]	0.498[D]	100.0	50	0.470[D]	0.636
Cadmium (Cd)	<0.005[U]	<0.005[U]	1.0	0.5	<0.005[U]	0.005^
Chromium (Cr)	<0.005[U]	<0.005[U]	5.0	2.5	<0.005[U]	0.050*
Lead (Pb)	<0.0075[U]	<0.0075[U]	5.0	2.5	<0.0075[U]	0.008*
Mercury (Hg)	0.0016[B]	0.0015[B]	0.2	0.1	0.0036	0.017
Selenium (Se)	<0.025[UJ]	<0.025[UJ]	1.0	0.5	0.0319[B]	0.047*
Silver (Ag)	<0.010[U]	<0.010[U]	5.0	2.5	<0.010[U]	0.010[^]
Underlying Hazardous Constituents (UHCs)						
Antimony (Sb)	<0.025[U]	<0.025[U]	-	-	<0.025[U]	0.0250[^]
Beryllium (Be)	<0.005[U]	<0.005[U]	-	-	<0.005[U]	0.007*
Nickel (Ni)	<0.005[U]	<0.005[U]	-	-	<0.005[U]	0.018*
Thallium (Tl)	<0.005[UD]	<0.005[UD]	-	-	<0.005[UD]	0.005^
Select Solids Analyses of Regulatory Interest						
	(mg/kg)	(mg/kg)			(mg/kg)	(mg/kg)
Benzene	<0.00098[U]	<0.00098[U]	-	-	<0.001[U]	0.00092[+]
Amenable Cyanide	<0.214[U]	1.80	-	-	<0.198[UL]	2.54*
Total Cyanide	11.8	21.6[D]	-	-	11.9	11.0
Phenol	<0.937[UJ]	2.67[J]	-	-	<0.741[UJ]	0.807[+]

-Indicates a location in the table for which an entry would not be appropriate.

[U] Non-detected analyte

[L] Sample result was more negative than the reporting limit.
[D] Results reported from a dilution.
[J] Matrix spike and/or matrix spike duplicate criteria was not met.
[B] Analyte was detected at the instrument at or above Limits of Detection (LOD), but less than Limit of Quantitation (LOQ).
***** Contains qualifier of "U" in at least one quarter.
[+] Contains qualifier of "U" in all quarters with multiple Reporting Limits (RL) or Limits of Detection (LOD).
^ Contains qualifier of "U" in all quarters with same RL or LOD.

Table 3-2. Mercury Speciation Data from Past Tank 50 Salt Solutions

Tank 50 Sample	Total Hg (mg/L)	Methyl Hg (mg/L)	Ratio Methyl Hg/Total Hg
2QCY17	72.2	32.2	0.446
3QCY17	81.4	28.2	0.346
1QCY18	71.8	36.6	0.510
2QCY18	69.8	28.5	0.408
3QCY18	70.4	30.7	0.436
4QCY18	61.7	18.8	0.305
1QCY19	67.4	24.0	0.356
2QCY19	63.0	19.3	0.306
3QCY19	67.6	19.9	0.294

Table 3-3 provides comparison between analytical results for each analyte to SwRI®'s Limit of Detection (LOD) and Limit of Quantitation (LOQ) for the TCLP leachates and to the Reporting Limits (RL) for the solids analyses. Antimony, arsenic, beryllium, cadmium, chromium, lead, nickel, selenium, silver and thallium were all less than the detection limit or reporting limit for the Full Premix sample. The same was true for the Cement-Free. Appendix A includes summaries of results from blanks, laboratory control samples, matrix spikes, and matrix spike duplicates.

Table 3-3. RCRA Metal TCLP Result Concentrations, Limit of Detection, and Limit of Quantitation[15]

Analyte	Methods	LOD	LOQ	Full premix Sample Results	Cement Free Sample Results
		(µg/L)	(µg/L)	(µg/L)	(µg/L)
Antimony (Sb)	6010D	25.0	50.0	<25.0[U]	<25.0[U]
Arsenic (As)	6010D	25.0	50.0	<25.0[U]	<25.0[U]
Barium (Ba)	6010D	50.0	100.0	450[D]	498[D]
Beryllium (Be)	6010D	5.00	10.0	<5.00[U]	<5.00[U]
Cadmium (Cd)	6010D	5.00	10.0	<5.00[U]	<5.00[U]
Chromium (Cr)	6010D	5.00	10.0	<5.00 [U]	<5.00[U]
Lead (Pb)	6010D	7.50	15.0	<7.50[U]	<7.50[U]
Mercury (Hg)	7470A	1.00	2.00	1.64[B]	1.46[B]
Nickel (Ni)	6010D	5.00	10.0	<5.00[U]	<5.00[U]
Selenium (Se)	6010D	25.0	50.0	<25.0[UJ]	<25.0[UJ]
Silver (Ag)	6010D	10.0	20.0	<10.0[U]	<10.0[U]
Thallium (Tl)	6020B	5.00	10.0	<5.00[UD]	<5.00[UD]
-	-	-	RL (mg/kg)	(mg/kg)	
Benzene	8260C	-	-	<0.00098[U]	<0.00098[U]
Amenable Cyanide	Amenable cyanide 9012B	-	0.214	<0.214[U]	1.80
Total Cyanide	Cyanide 9012B	-	0.249	11.8	21.6[D]
Phenol	Phenol 9065	-	0.937	<0.937[UJ]	2.67[J]

- Indicates a location in the table for which an entry would not be appropriate.
[U] Result is less than the Limit of Detection (LOD) and/or Reporting Limit (RL).
[D] Result is reported from a dilution.
[J] Matrix spike and/or matrix spike duplicate criteria was not met.
[B] Analyte was detected at the instrument at or above Limits of Detection (LOD), but less than Limit of Quantitation (LOQ).

4.0 Conclusions

Analyses of the SDF Full Premix and Cement-Free waste forms prepared from the 3QCY19 Tank 50 salt solution sample and premix material resulted in the following findings.

- The RCRA metal TCLP result concentrations met the SC Code of Regulations 61-79.261.24 requirements for a nonhazardous waste form.[6]
- The measured concentrations of the TCLP RCRA metals and additional inorganic/organic UHCs met the SC Code of Regulations 61-79.268.48 non-wastewater standards.[5]
- The measured concentrations of the TCLP RCRA metals met the SPF WAC.[7]

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Appendix A. Quality Assurance

The following subsections include summaries of results from blanks, laboratory control samples, matrix spikes, and matrix spike duplicates. The data package also includes data for calibration verifications, interference checks and serial dilutions.[15]

Table A- 1 shows all TCLP extraction fluid blank concentrations and the solid matrix blank concentrations. In the extraction fluid blank, antimony, arsenic, barium, beryllium, cadmium, chromium, lead, mercury, nickel, selenium, silver and thallium were all less than detection limit or reporting limit. Thallium was detected at the instrument at or above the LOD, but less than the LOQ. Benzene, amenable cyanide, total cyanide and phenol were all less than detection limit or reporting limit.

Table A- 1. TCLP Extraction Fluid Blank and Solid Matrix Blank[15]

Analyte	TCLP Blank (µg/L)	Qualifiers
Antimony (Sb)	<25.0	U
Arsenic (As)	<25.0	U
Barium (Ba)	<5.00	U
Beryllium (Be)	<5.00	U
Cadmium (Cd)	<5.00	U
Chromium (Cr)	<5.00	U
Lead (Pb)	<7.50	U
Mercury (Hg)	<0.100	U
Nickel (Ni)	<5.00	U
Selenium (Se)	<25.0	U
Silver (Ag)	<10.0	U
Thallium (Tl)	<5.00	UD
Analyte	Solid Matrix Blank (mg/Kg)	Qualifiers
Benzene	<0.00050	U
Amenable Cyanide	<0.249	U
Total Cyanide	<0.249	U
Phenol	<1.0	U

[U] Result is less than the Limit of Detection (LOD) and/or Reporting Limit (RL).
[D] Result is reported from a dilution.

Table A- 2 shows all LCS recoveries meet SwRI®'s acceptance limit in the range of 80% to 120% for metals and phenol, 70% to 130% for benzene and 95.7% for total cyanide, which was within the manufacturers acceptance limit.[17] The laboratory control samples are clean aqueous solutions analyzed to assure integrity of the analytical technique exclusive of matrix effects.

Table A- 2 Laboratory Control Sample[15]

Analyte	Laboratory Control (µg/L)		Recovery (%)
	True	Found	
Antimony (Sb)	500	473	94.6%
Arsenic (As)	2000	1900	95.0%
Barium (Ba)	2000	1890	94.5%
Beryllium (Be)	50.0	53.0	106.0%
Cadmium (Cd)	50.0	46.2	92.4%
Chromium (Cr)	200	183	91.5%
Lead (Pb)	500	444	88.8%
Mercury (Hg)	1.00	1.02	102.0%
Nickel (Ni)	500	449	89.8%
Selenium (Se)	2000	1760	88.0%
Silver (Ag)	50.0	48.0	96.0%
Thallium (Tl)	2000	1890	94.5%
Analyte	**Laboratory Control (mg/Kg)**		**Recovery (%)**
	True	**Found**	
Benzene	0.010	0.0097	97.0%
Amenable Cyanide	-	-	-
Total Cyanide	42.2	40.4	95.7%
Phenol	25.0	25.0	100.0%

- Indicates a location in the table for which an entry would not be appropriate.

Results from analysis of the matrix spike (MS) and the matrix spike duplicate (MSD) are given in Table A-3 and Table A-4. These results shown in Table A- 3 indicate all analytes met the recommended quality control acceptance criteria for MS and MSD percent recoveries (75-125%) and the Relative Percent Difference (RPD) acceptance limits (0-20%). In Table A-4, results show benzene met the recommended quality control acceptance criteria for MS, MSD and RPDs. In Table A-4, results show total cyanide and phenol did not meet the recommended quality control acceptance criteria for MS, MSD and RPDs. However, a post-digestion spike recovery sample showed a phenol recovery ≥ 75% within the control limit of 60% to 120%.

Table A- 3 TCLP Leachates Matrix Spike and Duplicate Results[15]

Analyte	Initial Concentrations (µg/L)			Spiked Sample** (µg/L)		Recovery (%)		RPD (%)
	Parent Sample Result	Qualifiers	Spike Added	Spike	Spike Duplicate	Spike	Spike Duplicate	
Antimony (Sb)	<25.0	U	5000	4830	4930	96.6	98.6	2.0
Arsenic (As)	<25.0	U	2500	2430	2440	97.2	97.6	0.4
Barium (Ba)	450	D	5000	5170	5250	94.4	96.0	1.7
Beryllium (Be)	<5.00	U	500	487	481	97.4	96.2	1.2
Cadmium (Cd)	<5.00	U	500	430	429	86.0	85.8	0.2
Chromium (Cr)	<5.00	U	1000	860	861	86.0	86.1	0.1
Lead (Pb)	<7.50	U	2500	2140	2110	85.6	84.4	1.4
Mercury (Hg)	1.6	B	10.0	11.3	11.1	96.6	94.6	2.1
Nickel (Ni)	<5.00	U	2500	2150	2150	86.0	86.0	0.0
Selenium (Se)	<25.0	UJ	2500	2350	2350	94.0	94.0	0.0
Silver (Ag)	<10.0	U	500	427	435	85.4	87.0	1.9
Thallium (Tl)	<5.00	UD	2500	2180	2190	87.2	87.6	0.5

[U] Result is less than the Limit of Detection (LOD) and/or Reporting Limit (RL).
[D] Result is reported from a dilution.
[B] Analyte was detected at the instrument at or above Limits of Detection (LOD), but less than Limit of Quantitation (LOQ).
[J] Matrix spike and/or matrix spike duplicate criteria was not met.
** SwRI® Sample ID = W-18193-10001 MS/MSD

Table A-4. Organic UHCs Matrix Spike and Duplicate Results[15]

Analyte	Initial Concentrations (mg/kg)				Spiked Sample (mg/kg)		Recovery (%)		RPD (%)
	Result	Qualifiers	MS-Spike Added	MSD-Spike Added	Spike	Spike Duplicate	Spike	Spike Duplicate	
Benzene*	0.0	U	0.020	0.020	0.017	0.016	85	80	6.0
Amenable Cyanide	-	-	-	-	-	-	-	-	-
Total Cyanide***	11.8**	-	1.72	1.59	15.6	15.5	220.9	232.7	5.2
Phenol***	0.937	UJ	22.8	22.5	0.910	2.18	0.0	9.7	200.0

[U] Result is less than the Limit of Detection (LOD) and/or Reporting Limit (RL).
[J] Matrix spike and/or matrix spike duplicate criteria was not met.
* SwRI® Sample ID = W-18193-10001
**Parent value exceeded 4 times the spike added; therefore, MS/MSD %Recovery and %RPD are not required for evaluation
- Indicates a location in the table for which an entry would not be appropriate.
*** SwRI® Sample ID = W-18193-10001 MS/MSD

Distribution:

M. R. Alexander	K. B. Martin
J. P. Arnold	J. J. Mayer
C. J. Bannochie	M. W. McCoy
M. J. Barnes	R. T. McNew
M. N. Borders	D. J. McCabe
J. M. Bricker	G. A. Morgan
K. M. Brotherton	P. W. Norris
L. W. Brown	J. E. Occhipinti
N. F. Chapman	F. M. Pennebaker
J. H. Christian	R. C. Player
W. A. Condon	J. Polk
A. D. Cozzi	P. A. Polk
C. L. Crawford	M. M. Potvin
J. Crenshaw	A. A. Ramsey
D. A. Crowley	W. G. Ramsey
K. D. Dixon	J. W. Ray
R. E. Edwards	C. Ridgeway
A. P. Fellinger	L. B. Romanowski
S. D. Fink	K. H. Rosenberger
E. J. Freed	A. Samadi-Dezfouli
S. J. Harrington	D. C. Sherburne
E. W. Harrison	F. M. Smith
C. C. Herman	A. V. Staub
K. A. Hill	J. Stevens
P. J. Hill	M. E. Stone
T. H. Huff	P. C. Suggs
J. F. Iaukea	B. J. Wiedenman
V. Jain	T. L. White
C. A. Langton	A. W. Wiggins
J. D. Ledbetter	L. A. Wooten
K. R. Liner	R. H. Young
M. J. Mahoney	Records Administration (EDWS)
J. Manna	